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Indian Standard

SPECIFICATION FOR HEXAMETHYLENETETRAMINE (HEXAMINE)

(Second Revision)

UDC 661·7 : 547·288·15

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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May 1988

AMENDMENT NO. 1 (FEBRUARY 1990
TO
IS 4306 : 1987 SPECIFICATION FOR HEXAMETHYLENETETRAMINE (HEXAMINE)

(Second Revision)

(*Page* 1, *clause* 0.4) Substitute ' 2.1.2 ' *for* ' 3.1.3 '.

(*Page* 2, *clause* 2.1.1) — Delete.

(PCD 9)

Reprography Unit, BIS, New Delhi

Indian Standard

SPECIFICATION FOR HEXAMETHYLENETETRAMINE (HEXAMINE)

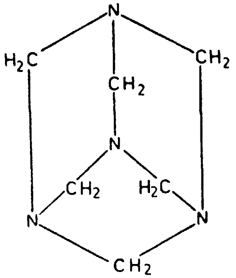
(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 30 October 1987, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1967 and revised in 1973 incorporating the requirements and methods of determination of sodium compounds, angle of slip and free formaldehyde content, when ammonia is absent, for the materials used for the manufacture of high explosives. In the present version, the requirements for the explosives grade of the material have been excluded, the requirement of *p*H has been modified and potentiometric titration method for assay has been introduced for the product used for non-explosive purposes (*see* **1.1**). It is envisaged that the requirements for hexamine used for the manufacture of high explosives may be covered in a separate standard under the purview of Explosives and Pyrotechnics Sectional Committee.

0.3 Hexamethylenetetramine ($C_6H_{12}N_4$), also known as methenamine, urotropine and hexamine, is the raw material for high explosives like RDX and is also used in the manufacture and curing of phenolic resins, as a rubber vulcanization accelerator; as fuel tablets for camping stoves, as absorber of poisonous gases, and as reagent for the detection of gold, mercury, antimony, bismuth and silver. It is also used for pharmaceutical purposes, the requirements for which shall be as given in Indian Pharmacopoeia and the others recognized by the *Drugs and Cosmetics Act* and Rules. It is represented by the following structural formula:



Hexamethylenetetramine
(Hexamine)
(Molecular Mass 140·18)

0.4 This standard contains **3.1.3** which permits the purchaser to use his option for selection to suit his requirements.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960[*]. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for hexamethylenetetramine (hexamine) for non-explosive purposes.

2. REQUIREMENTS

2.1 Description — The material shall be colourless, lustrous crystals or a white crystalline free flowing powder, odourless, taste at first sweetish but afterwards bitter.

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[*] Rules for rounding off numerical values (*revised*).

2.1.1 The material shall sublime at a temperature of not less than 260°C with partial decomposition and evolution of a disagreeable odour. It shall burn readily with a blue smokeless flame.

2.1.2 The material shall be soluble in water, rectified spirit and chloroform.

2.1.3 The size of the material shall be as agreed to between the purchaser and the supplier.

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in polyethylene lined bags or in such other containers as agreed to between the purchaser and the supplier.

3.1.1 The polyethylene sheet shall not be less than 0·05 *mm in* thickness.

3.2 Marking — The containers shall be securely closed and legibly and indelibly marked with the following:

- a) Manufacturer's name and recognized trade-mark, if any;
- b) Name and net mass of the material in the container; and
- c) Lot or batch number, in code or otherwise.

3.2.1 The container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in Appendix B.

TABLE 1 REQUIREMENTS FOR HEXAMETHYLENETETRAMINE (HEXAMINE)

(*Clause* 2.2)

SL No.	CHARACTERISTICS	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), *Min*	99·0	A-2
ii)	*p*H of aqueous extract	8·0 to 9·0	A-3
iii)	Water insolubles, percent by mass, *Max*	0·05	A-4
iv)	Loss on drying, percent by mass, *Max*	0·5	A-5
v)	Sulphated ash, percent by mass, *Max*	0·1	A-6
vi)	Ammonia (as NH_3), percent by mass, *Max*	0·01	A-7
vii)	Arsenic (as As_2O_3), ppm, *Max*	2	A-8
viii)	Chloride (as Cl), percent by mass, *Max*	0·02	A-9
ix)	Sulphate (as SO_4), percent by mass, *Max*	0·005	A-10
x)	Iron (as Fe), percent by mass, *Max*	0·005	A-11
xi)	Heavy metals (as Pb), ppm, *Max*	10	A-12
xii)	Organic impurities	To pass test	A-13

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR HEXAMETHYLENETETRAMINE (HEXAMINE)

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. ASSAY

A-2.0 Outline of the Method — The known quantity of material is dissolved in glacial acetic acid and the solution is potentiometrically titrated with standard perchloric acid while stirring and potential is measured using a combined glass calomel electrode. Equivalence point is determined where an abrupt change in potential is noted.

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*Specification for water for general laboratory use (*second revision*).

A-2.1 Reagents

A-2.1.1 *Glacial Acetic Acid*

A-2.1.2 *Perchloric Acid* — N/1.

A-2.2 Apparatus

A-2.2.1 *Beaker* — 250 ml.

A-2.2.2 *Combined Glass Calomel Electrode*

A-2.2.3 *Burette*

A-2.3 Procedure — Weigh accurately about 5 g of the material and transfer it to a 250-ml tall form beaker. Dissolve it in 100 ml glacial acetic acid. Insert a stirring bar in the beaker. Submerge the active surface of the electrodes in the contents. Measure the potential in mV before any titrant is added. Record this value and the initial burette reading.

A-2.3.1 In the beginning of the titration, add the titrant (**A-2.1.2**) in 1 ml increment when the mV is changing slowly, but near the expected end point add the titrant in increments of 0·1 ml. Continue the titration until an abrupt change in potential is noted. Record the potential for three to four small increments of titrant beyond the end point.

A-2.4 Calculation — Make a table of the observations before and after the end-point, and calculate the total volume of titrant used at the end-point as indicated below:

Table of Observations		*Calculation*
Titrant Added (ml)	Meter Reading	
v_1	E_1	
$v_2 = v_1 + d$	E_2	$\Delta_1 E = E_1 - E_2$
$v_3 = v_1 + 2d$	E_3	$\Delta_2 E = E_2 - E_3$ $\Delta_1(\Delta E) = \Delta_1 E - \Delta_2 E$
$v_4 = v_1 + 3d$	E_4	$\Delta_3 E = E_3 - E_4$ $\Delta_2(\Delta E) = \Delta_2 E - \Delta_3 E$
$v_5 = v_1 + 4d$	E_5	$\Delta_4 E = E_4 - E_5$ $\Delta_3(\Delta E) = \Delta_3 E - \Delta_4 E$
$v_6 = v_1 + 5d$	E_6	$\Delta_5 E = E_5 - E_6$ $\Delta_4(\Delta E) = \Delta_4 E - \Delta_5 E$

The equivalence point is at v_e ml of titrant, in which

$$v_e = v_3 + \frac{\Delta_2(\Delta E)}{\Delta_2(\Delta E) - \Delta_3(\Delta E)} \times d$$

where

v_1, v_2, v_3, v_4, v_5 and v_6 = subsequent burette readings;

d = equal and small volume of titrant added; and

E_1, E_2, E_3, E_4, E_5, and E_6 = subsequent meter reading at the burette readings mentioned.

The assay is calculated as in **A-2.4.1**.

A-2.4.1 *Expression of Results*

Assay, percent by mass

$$= \frac{V \times N \times 0{\cdot}140\,1 \times 100}{M}$$

where

V = volume in ml of standard perchloric acid solution used for titration of the sample,

N = normality of perchloric acid solution, and

M = mass in g of the sample taken.

A-3. DETERMINATION OF *p*H

A-3.1 Apparatus

A-3.1.1 *p*H *Meter* — fitted with glass electrode.

A-3.2 Procedure — Weigh accurately 10 g of the material, dissolve in 100 ml of freshly boiled and cooled distilled water. Determine the *p*H by means of a *p*H-meter using glass electrode.

A-4. DETERMINATION OF WATER INSOLUBLES

A-4.1 Procedure — Weigh accurately about 20 g of the material in a beaker and dissolve it in 250 ml of water. Filter the insolubles in a tared sintered glass crucible (C_3). Wash the residue with successive portions of distilled water. Dry the crucible at 105 ± 2°C, cool and weigh. Repeat drying and weighing till two subsequent weighings do not differ by more than 0·001 g.

A-4.2 Calculation

Water insolubles, percent by mass

$$= \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue after drying, and

M_2 = mass in g of the material taken for the test.

A-5. DETERMINATION OF LOSS ON DRYING

A-5.1 Procedure — Weigh accurately about 5 g of the material in a tared glass petri dish and dry the material in a desiccator over sulphric acid or dry silica gel till constant mass.

A-5.2 Calculation

Loss on drying, percent by mass

$$= \frac{M_1 \times 100}{M_2}$$

where

M_1 = loss in mass in g of the material, and

M_2 = mass in g of the material taken for the test.

A-6. DETERMINATION OF SULPHATED ASH CONTENT

A-6.0 Outline of the Method — A known quantity of the material is carbonized, treated with sufficient sulphuric acid, then incinerated and total ash determined.

A-6.1 Procedure — Take about 2 to 3 g, accurately weighed, of the ground material in a tared platinum, silica or porcelain dish, previously ignited and weighed. Ignite until thoroughly charged, cool, moisten the residue with 1 ml of sulphuric acid, then cautiously ignite until the carbon is completely consumed. Conduct the ignition in a place protected from air currents using as low a temperature as possible to effect the combustion of all the carbon and then heat strongly till no fumes are evolved. When the carbon has completely disappeared, cool the crucible in a desiccator, and weigh. Calculate the percentage of sulphated ash with reference to the material taken for the test.

A-6.2 Calculation

Sulphated ash, percent by mass

$$= \frac{(M_1 - M_2)}{M} \times 100$$

where

M_1 = mass in g of the ignited residue with the crucible,

M_2 = mass in g of the crucible, and

M = mass in g of the material taken for the test.

A-7. DETERMINATION OF AMMONIA CONTENT

A-7.0 Outline of the Method — The colour produced by the material with Nessler's reagent is compared to that produced by standard ammonia solution under identical conditions.

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50-ml capacity (*see* IS : 4161-1967*).

A-7.2 Reagents

A-7.2.1 *Potassium Iodide* — crystalline, free from iodate.

A-7.2.2 *Mercuric Chloride Solution* — saturated.

A-7.2.3 *Potassium Hydroxide* — Prepare 9 molar solution or dissolve 126·2 g of solid potassium hydroxide in 250 ml of water free from ammonia.

A-7.2.4 *Nessler's Reagent* — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water and add to it slowly with stirring mercuric chloride solution, until a slight permanent precipitate is formed. Then add 80 ml of potassium hydroxide solution, add 1 ml more of the mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight. Decant the clear solution and keep the solution in a bottle closed with a well-fitting rubber stopper.

A-7.2.5 *Standard Ammonia Solution* — Dissolve 3·141 g of ammonium chloride in ammonia-free water and make up the volume to 1 000 ml. Dilute 100 ml of this solution to 1 000 ml with ammonia-free water. One millilitre of this solution contains 0·1 mg of ammonia (as NH_3).

A-7.3 Procedure — Take 10 ml of the 10 percent (m/v) solution of the material into a Nessler cylinder and dilute with 30 ml of water. Then add 2 ml of Nessler's reagent. Make up the volume to 50 ml. Carry out a control test in the other Nessler cylinder, using 1 ml of standard ammonia solution and 2 ml of Nessler's reagent in the same total volume of the reaction mixture Compare the colour produced in the two cylinders after 5 minutes.

A-7.3.1 The limit prescribed for ammonia shall be taken as not having been exceeded if any colour produced in the test with the material is not deeper than that produced in the control test.

A-8. DETERMINATION OF ARSENIC CONTENT

A-8.0 Outline of the Method — The stain produced by arsenic on a mercuric bromide paper is compared to stains produced by standard arsenic solution.

A-8.1 Procedure — Weigh accurately 5 g of the material and dissolve in distilled water. Make up the volume to 100 ml in a volumetric flask. Take 50 ml of this solution in the Gutzeit bottle and proceed as described in IS : 2088-1983*, using for comparison 5 ml of standard arsenic trioxide solution, 1 ml of which contains 0·001 mg of arsenic trioxide (as As_2O_3).

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*Specification for Nessler cylinders.

*Method for determination of arsenic (*second revision*).

A-9. DETERMINATION OF CHLORIDE CONTENT

A-9.0 Outline of the Method — The opalescence produced by the standard chloride solution with silver nitrate solution is compared to that produced by the material under identical conditions.

A-9.1 Apparatus

A-9.1.1 *Nessler Cylinders* — same as described in **A-7.1.1**.

A-9.2 Reagents

A-9.2.1 *Dilute Nitric Acid* — approximately 5 N.

A-9.2.2 *Silver Nitrate Solution* — approximately 5 pertent (*m/v*).

A-9.2.3 *Standard Sodium Chloride Solution* — Dissolve 0·164 9 g of sodium chloride in water and make up the volume to 1 000 ml. One millilitre of this solution contains 0·1 mg of chloride (as Cl).

A-9.3 Procedure — Take 10 ml of the 10 percent (*m/v*) solution of the material into a Nessler cylinder and dilute with 30 ml of water. Then add 6 ml of dilute nitric acid and 1 ml of silver nitrate solution. Make up the solution to 50 ml with water. Carry out a control test in the other Nessler cylinder, using 2 ml of standard chloride solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the opalescence produced in the two cylinders after 5 minutes.

A-9.3.1 The limit prescribed for chlorides shall be taken as not having been exceeded if the opalescence produced in the test with the material is not greater than that produced in the control test.

A-10. DETERMINATION OF SULPHATE (SO_4) CONTENT

A-10.0 Outline of the Method — The turbidity produced by the material with barium chloride is compared to that produced by the standard sulphate solution under identical conditions.

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — same as described under **A-7.1.1**.

A-10.2 Reagents

A 10.2.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-10.2.2 *Barium Chloride Solution* — approximately 10 percent (*m/v*).

A-10.2.3 *Standard Sulphate Solution* — Dissolve 0·181 4 g of potassium sulphate in water and make up the volume to 1 000 ml. One millilitre of this solution contains 0·1 mg of sulphate (as SO_4).

A-10.3 Procedure

A 10.3.1 Take 20 ml of 10 percent (*m/v*) solution of the material, add 15 ml of dilute hydrochloric acid and evaporate to dryness on a steam-bath. Dissolve the residue in 15 ml of water and transfer into a Nessler cylinder washing the dish thoroughly with water. Add 2 drops of dilute hydrochloric acid and 2 ml of barium chloride solution. Dilute the resulting solution with water to 50 ml mark. Carry out a control test in the other Nessler cylinder using 1 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidity produced in the two cylinders after 20 minutes.

A-10.3.2 The limits prescribed for sulphates shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-11. DETERMINATION OF IRON

A-11.0 Outline of the Method — The colour produced in the butanolic layer, when the material is treated with hydrochloric acid, ammonium persulphate and butanolic potassium thiocyanate, is compared to that produced by the standard iron solution under identical conditions.

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinder* — same as described under **A-7.1.1**.

A-11.2 Reagents

A-11.2.1 *Hydrochloric Acid* — sp gr 1·16 (*see* IS : 265-1976*).

A-11.2.2 *Ammonium Persulphate* — solid.

A-11.2.3 *Butanolic Potassium Thiocyanate* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-11.2.4 *Dilute Sulphuric Acid* — approximately 10 percent (*m/v*).

A-11.2.5 *Standard Iron Solution* — Weigh 0·863 5 g of ferric ammonium sulphate [$F_eNH_4(SO_4)_2.12H_2O$] and dissolve in 10 ml of dilute sulphuric acid. Dilute with water to make up the volume to 1 000 ml. Transfer 10

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*Specification for hydrochloric acid (*second revision*).

10 ml of this solution and again dilute with water to make up the volume to 100 ml. One millilitre of this solution is equivalent to 0·01 mg of iron.

A-11.3 Procedure

A-11.3.1 Take 10 ml of the 10 percent (*m/v*) solution of the material and make acidic with hydrochloric acid. Boil for one minute and cool to room temperature. Add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocynate. Shake vigorously for 30 seconds and allow the liquid to separate. Carry out a control test using 5 ml of the standard iron solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the butanol layer after 2 minutes.

A-11.3.1.1 The limit prescribed for iron shall be taken as not having been exceeded if any red colour produced in the test with the material is not darker than that produced in the control test.

A-12. DETERMINATION OF HEAVY METAL CONTENT

A-12.0 Outline of the Method—The colour produced by heavy metals with hydrogen sulphide is compared to that produced by standard lead solution under identical conditions.

A-12.1 Apparatus

A-12.1.1 *Nessler Cylinders* —Same as described uuder **A-7.1.1.**

A-12.2 Reagents

A-12.2.1 *Dilute Acetic Acid* — 1 : 1. Dilute acetic acid which complies with the following additional test:

Evaporate 20 ml of the acid in porcelain dish nearly to dryness on a steam-bath. Add to the residue 2 ml of dilute acetic acid and dilute with water to 25 ml. Then add 10 ml of a solution of hydrogen sulphide. Any dark colour produced shall not be darker than a control made with 0·04 mg of lead and 2 ml of the diluted acetic acid (2 ppm).

A-12.2.2 *Hydrochloric Acid* — Hydrochloric acid which complies with the following addition test:

Evaporate 17 ml of the acid in a beaker to dryness on a steambath. Dissolve the residue in 2 ml of dilute acetic acid and dilute with water to 40 ml. Then add 10 ml of hydrogen sulphide solution. Any dark colour produced shall not be greater than a control prepared from 0·02 mg of lead and 1 ml of dilute acetic acid (1 ppm).

A-12.2.3 *Hydrogen Sulphide Solution* — a freshly prepared saturated solution of hydrogen sulphide in water.

A-12.2.4 *Stock Lead Nitrate Solution* — Dissolve 159·8 mg of lead nitrate in 100 ml of water to which 1 ml of nitric acid has been added, then dilute to 1 000 ml with water.

NOTE — This solution shall be prepared and stored in glass containers free from soluble lead salts.

A-12.2.5 *Standard Lead Solution* — Dilute 10 ml of the stock solution of lead nitrate accurately to 100 ml with water. This solution shall be freshly prepared. One millilitre of this standard lead solution contains the equivalent of 0·01 mg of lead. When 1 ml of the standard lead solution is employed to prepare the solution to be compared with a solution of 1 g of the substance being tested, the comparison solution thus prepared contains the equivalent of 10 parts of lead per million parts of the substance being tested.

A-12.2.6 *Test Solution*

Solution A — Introduce into a 50-ml Nessler cylinder 2 ml of dilute acetic acid and 1 ml of standard lead solution containing the lead equivalent of the heavy metals limit of 10 ppm, and make up the volume to 25 ml with water.

Solution B — Dissolve 1 g of the material under test in 10 ml of water, add 2 ml of dilute hydrochloric acid and water to make up the volume to 25 ml.

A-12.3 Procedure — Transfer Solutions *A* and *B* to matching 50-ml Nessler cylinder, add 10 ml of solution of hydrogen sulphide to each cylinder; mix, allow to stand for 10 minutes, then view downward over a white surface; the colour of *Solution B* shall not be darker than that of *Solution A*.

A-13. TEST FOR ORGANIC IMPURITIES

A-13.1 Reagent

A-13.1.1 *Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1977*).

A-13.2 Procedure — Dissolve 0·1 g of the material, dried in a desiccator over sulphuric acid, in 2 ml concentrated sulphuric acid. Cool the solution because the reaction is vigorous in warm condition. The sample shall be taken to comply with the standard if the solution is clear and colourless.

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*Specification for sulphuric acid (*second revision*).

APPENDIX B

(*Clause* 4.1)

SAMPLING OF HEXAMETHYLENETETRAMINE (HEXAMINE)

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B 1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.3 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.4 The sample containers shall be of such a size that they are almost completely filled by sample.

B-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment of the material, all the containers of the same size and drawn from a single batch of manufacture, shall constitute a lot. If a consignment of the material is known to consist of different sizes of the containers or different batches of manufacture, the containers belonging to the same size and batch shall be grouped together and each group shall constitute a lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (n) shall depend on the size of the lot (N) and shall be in accordance with Table 2.

B-2.3 The containers shall be selected at random from the lot and to ensure randomness of selection, random number tables shall be used.

In case, such tables are not available, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3, ..., etc, up to r, and so on, where r is the integral part of N/n (N being the number of containers in the lot and n the number of containers to be selected). Every rth container thus counted shall be withdrawn to give sample for tests.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clause* B-2.2)

Lot Size N (1)	No. of Containers to be Selected n (2)
4 to 25	3
26 ,, 50	4
51 ,, 100	5
101 ,, 150	6
151 ,, 300	7
301 and above	8

Note — When the size of the lot is three or less, all the containers shall be sampled.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a representative portion of the material shall be taken out with the help of a suitable sampling instrument. The total quantity so drawn from each of the container shall be approximately equal to thrice the quantity required for testing purposes.

B-3.2 Out of these portions, an equal quantity of the material shall be taken and thoroughly mixed to form a composite sample of about 600 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the n containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.4 All the individual and composite samples shall be transferred to separate containers. These containers shall be sealed air-tight and labelled with full identification particulars given in **B-1.5**.

B-3.5 The referee test samples consisting of a composite sample and a set of *n* individual samples shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier. and should be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Test for assay shall be performed on the individual samples.

B.4,2 Tests for the determination of all other characteristics given in 2 and Table 1 shall be performed on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the test results for assay, the mean (X) and range (R) of test results shall be computed (range being defined as the difference between the maximum and minimum values of test results).

B-5.1.1 The lot shall be declared as conforming to the requirements of assay if the value of the expression ($X - 0{\cdot}6\ R$) is greater than or equal to the relevant limits specified in Table 1.

B-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result of each of the characteristics shall satisfy the relevant requirements specified.

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